Chemistry Department
Christ Church Grammar School

Year 12
2022

Volumetric Analysis/Titration Validation Test

Time allowed: 45 minutes

Instructions
Please ensure you enter your name and circle your teacher's initials below.
Scientific calculators only.
Chemistry Data Sheet will be provided

Name
Solutions

Teacher: (circle)

BLR NMO MXC

Mark:____________ / **39**

Spare working paper on back page if required

Question 1 **25 marks**

A student wishes to determine the percentage of ammonia in a solution of household window cleaner by titrating a diluted solution of the window cleaner against hydrochloric acid. The student needs to standardise hydrochloric acid used before titrating it against the dilute ammonia solution. The following steps were used.

Step 1: Preparation of Primary Standard.

A 4.850 g sample of anhydrous sodium carbonate was dissolved and transferred into a 500.0 mL volumetric flask and made up to volume with distilled water.

(a) Determine the concentration of the **standard** sodium carbonate solution.

$M(Na_2CO_3) = 2 \times 22.99 + 12.01 + 3 \times 16.00 = 105.99 \text{ g mol}^{-1}$

$n(Na_2CO_3) = \frac{m}{M} = \frac{4.850}{105.99} = 0.045759 \text{ mol}$ (1)

$[Na_2CO_3] = \frac{n}{V} = \frac{0.045759}{0.500} = 0.0915 \text{ mol L}^{-1}$ (1)

(2 marks)

(b) (i) Sodium carbonate is used as a primary standard as it has a relatively high molar mass. Explain why it is important for a primary standard to have a relatively high molar mass.

To minimize the effect of uncertainty when the mass is measured.

(1 mark)

(ii) Give two additional reasons (excluding high molar mass and solubility) why sodium carbonate is a suitable compound to be used as a primary standard.

Reason 1 Readily obtainable in a pure form

Reason 2 Stores without deteriorating or reacting with atmosphere

(2 marks)

Known chemical formula

(Any 2 (1) mark each)

Step 2: Standarisation of the HCℓ solution.

An average titre volume of 14.9 (± 0.1) mL of the primary standard solution from Step 1 was used to neutralise a 20.00 (± 0.03) mL aliquot hydrochloric acid solution.

(d) Calculate the percentage uncertainty of the 20.00 mL aliquot of hydrochloric acid solution.

$\frac{0.03}{20.00} \times 100 = 0.15\%$ ie 20 mL ± 0.15 %

(1 mark)

(e) Calculate the percentage uncertainty of the average titre volume.

$\frac{0.1}{14.9} \times 100 = 0.67\%$ ie 14.9 mL ± 0.67%

(1 mark)

(f) Calculate the standardized concentration of HCℓ and the absolute uncertainty associated with the determined concentration. (Note: Assume the % uncertainty from the primary standard is negligible in this case.)

$V(Na_2CO_3) = 14.9 \times 10^{-3}$ L

$n(Na_2CO_3) = cV = 0.0915 \times 14.9 \times 10^{-3}$

$= 0.00136$ mol (1)

$n(HCl) = 2 \times n(Na_2CO_3)$

$= 2 \times 0.00136$

$= 0.00272$ mol (1)

$[HCl] = \frac{n}{V} = \frac{0.00272}{20.00 \times 10^{-3}}$

$= 0.136$ mol L^{-1} (1)

Total % uncertainty $= 0.15 + 0.67 = 0.82\%$ (1)

$\frac{0.82}{100} \times 0.136 = 0.001$

$\therefore [HCl] = 0.136 \pm 0.001$ mol L^{-1} (1)

(5 marks)

Question 1 continued

Step 3: Determination of the % ammonia in window cleaner.

A 20.00 mL sample of window cleaner, with a mass of 20.90 g, was transferred into a 250.00 mL volumetric flask and made up to the mark with distilled water. The diluted window cleaner (ammonia solution) was then transferred to a 50.00 mL burette and titrated against 25.00 mL aliquots of the standardised hydrochloric acid solution from Step 2.

The following results were obtained:

	Volume of diluted ammonia solution (mL)			
	Rough	1	2	3
Initial volume (mL)	0.50	1.20	2.55	1.30
Final volume (mL)	37.55	37.15	38.50	37.20
Titre volume (mL)	37.05	35.95	35.95	35.90

(Note: You do not need refer to uncertainties in part (g) and (h))

(g) Determine the average titre volume.

$\frac{35.95 + 35.95 + 35.90}{3} = 35.93$ mL

(1 mark)

(h) Determine the percentage of ammonia in the window cleaner solution.
(Note: if you were unable to determine the concentration of standardised HCℓ in part (e) on page 3, use [HCℓ] = 0.100 molL^{-1})

$V(HCl) = 25.00 \times 10^{-3}$ L

If using $[HCl] = 0.100$ molL^{-1} ⇓

$[HCl] = 0.136$ molL^{-1}

$n(HCl) = cV = 0.136 \times 25.00 \times 10^{-3}$

$= 0.0034$ mol (1) 0.0025 mol

$n(NH_3)$ in titre $= n(HCl)$

$= 0.0034$ mol (1) 0.0025 mol

$[NH_3]_{diluted} = \frac{n}{V} = \frac{0.0034}{35.93 \times 10^{-3}}$

$= 0.09463$ molL^{-1} (1) 0.06958 molL^{-1}

$n(NH_3)$ in 250 mL flask $= 0.09463 \times 0.250$

$= 0.02366$ mol (1) 0.017395 mol

$n(NH_3)$ in 20 mL undiluted = 0.02366 mol (1) 0.017395 mol

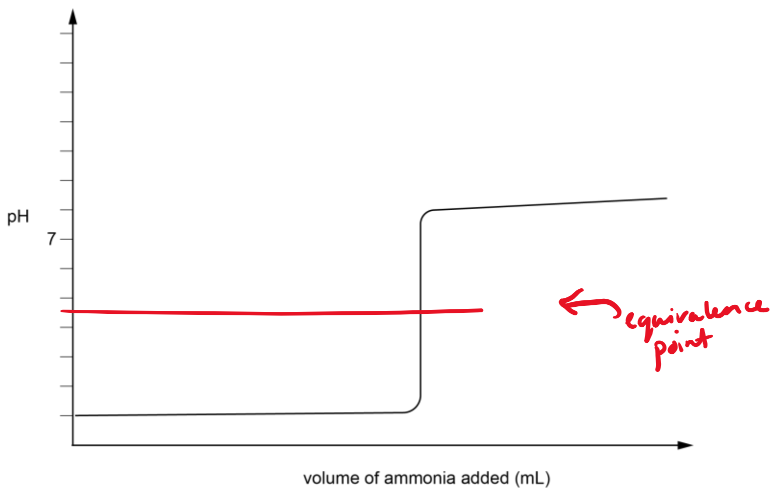
$m(NH_3) = n \times M$

$= 0.02366 \times (14.01 + 3 \times 1.008) = 0.403\ g$ (1) 0.296 g

$\% NH_3 = \frac{0.403}{20.90} \times 100 = 1.93\ \%$ (1) 1.42 %

(7 marks)

The titration curve of the reaction between the diluted ammonia solution and hydrochloric acid in Step 3 is shown below.



The student had 3 indicators to choose from for the titration in Step 3.

Indicator	pH range	Colour (lower pH – higher pH)
Methyl orange	3.1 – 4.4	red – yellow
Thymol blue	1.2 – 2.8	red – yellow
Phenolphthalein	8.3 – 10.0	colourless – pink

(g) Which indicator should they choose to identify the equivalence point?

methyl orange

(1 mark)

(f) Explain your choice of indicator, using appropriate chemical equations. Include the term 'equivalence point' and 'end point' in your response.

Reaction for titration $NH_3 + H_3O^+ \rightarrow NH_4^+ + H_2O$ (1)

NH_4^+ will undergo hydrolysis $NH_4^+ + H_2O \rightarrow NH_3 + H_3O^+$ (1)

$[H_3O^+]$ results in pH of ≈ 4.5 at equivalence point (1)

Methyl orange will change colour closest to this point enabling the end point to be identified closest to the equivalence point. (1)

(4 marks)

Question 2 **14 marks**

Marble is composed mainly of calcium carbonate, $CaCO_3$, with some impurities.

A 2.48 g sample of marble was dissolved in 500.0 mL of standardised 0.204 $molL^{-1}$ nitric acid. The mixture fizzed as carbon dioxide was produced and the marble completely dissolved. After the reaction mixture finished bubbling, samples of the remaining solution were transferred to a 50.00 mL burette and titrated against 20.00 mL aliquots of 0.100 $molL^{-1}$ NaOH solution. The average titre volume required was determined as 18.70 mL.

(a) Calculate the percentage by mass of calcium carbonate in the sample of marble.

$CaCO_3 + 2HNO_3 \rightarrow Ca(NO_3)_2 + CO_2 + H_2O$

$n(H^+)_{before} = cV = 0.204 \times 0.500 = 0.102$ mol (1)

$n(NaOH) = cV = 0.100 \times 20 \times 10^{-3} = 0.002$ mol (1)

$n(H^+)$ in titre $= n(NaOH) = 0.002$ mol (1)

$[H^+] = \frac{n}{V} = \frac{0.002}{18.70 \times 10^{-3}} = 0.10695\ molL^{-1}$ (1)

$n(H^+)_{after} = cV = 0.10695 \times 0.500 = 0.053475$ mol (1)

$n(H^+)_{reacted} = 0.102 - 0.053475 = 0.048525$ mol (1)

$n(CaCO_3)$ reacted $= \frac{n(HNO_3)}{2} = \frac{0.048525}{2} = 0.02426$ mol (1)

$m(CaCO_3) = n \times M = 0.02426 \times (40.08 + 12.01 + 3 \times 16.00)$

$= 2.428$ g (1)

$\% CaCO_3 = \frac{2.428}{2.48} \times 100 = 97.9\%$ (1)

(9 marks)

(b) State the effect of rinsing the following equipment with the solution given on the apparent % of calcium carbonate in the sample of marble.
Use "increase", "decrease" or "no change".

	Equipment	Rinsed with	Effect on apparent % of calcium carbonate in sample of marble
(i)	burette	distilled water	increase
(ii)	pipette	distilled water	decrease
(iii)	conical flask	distilled water	no change

(3 marks)

(c) Explain your response to part (b) (i)

Rinsing burette with distilled water could dilute H^+ in burette leading to a higher titre value and a lower determined value of $[H^+]$ remaining in sample. (1) This in turn would make it appear that more H^+ has reacted (1) leading to a higher apparent value of $CaCO_3$ in sample.

(2 marks)

END OF TEST

Spare working paper if required